(1390 REV. 5-93) US DEPT. OF COMMERCE PATENT & TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER
110486

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

U.S. APPLICATION NO. (if known, see 37 C.F.R.1.5)
09/914442

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/FI00/00221	March 17, 2000	March 18, 1999

TITLE OF INVENTION
PROCESS FOR PRODUCING A CROSS-LINKED POLYMER PRODUCT

APPLICANT(S) FOR DO/EO/US
Ali HARLIN, Matti HIRVENSALO

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.

2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.

3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).

4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.

5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
   a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☒ has been transmitted by the International Bureau.
   c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)

6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).

7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ have been transmitted by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   d. ☐ have not been made and will not be made.

8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).

9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).

10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.

12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.

13. ☒ A FIRST preliminary amendment.

    ☐ A SECOND or SUBSEQUENT preliminary amendment.

14. ☐ A substitute specification.

15. ☐ Entitlement to small entity status is hereby asserted.

16. ☒ Other items or information: Submission of the Annexes to the IPER

U.S. APPLICATION NO. (if known, see 37 C.F.R. 1.5)	INTERNATIONAL APPLICATION NO.	ATTORNEY'S DOCKET NUMBER
09/914442	PCT/FI00/00221	110486

17. ☒ The following fees are submitted:	CALCULATIONS	PTO USE ONLY
Basic National fee (37 CFR 1.492(a)(1)-(5)):		
Search Report has been prepared by the EPO or JPO $860.00		
International preliminary examination fee paid to USPTO (37 CFR1.482) $690.00		
No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) $710.00		
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO.....$1,000.00		
International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4).....$ 100.00		
ENTER APPROPRIATE BASIC FEE AMOUNT =	$860.00	
Surcharge of $130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(e)).	$	

Claims	Number Filed	Number Extra	Rate		
Total Claims	12- 20 =	0	X $ 18.00	$	
Independent Claims	1- 3 =	0	X $ 80.00	$	
Multiple dependent claim(s)(if applicable)			+ $270.00	$	
TOTAL OF ABOVE CALCULATIONS =				$	
Reduction by 1/2 for filing by small entity, if applicable.			-	$	
SUBTOTAL =				$860.00	
Processing fee of $130.00 for furnishing the English translation later than ☐ 20 ☐ 30 month from the earliest claimed priority date (37 CFR 1.492(f)).			+	$	
TOTAL NATIONAL FEE =				$860.00	
				Amount to be refunded	$
				Charged	$

a. ☒ Check No. 122158 in the amount of $860.00 to cover the above fees is enclosed.
b. ☐ Please charge my Deposit Account No. ___ in the amount of $___ to cover the above fees. A duplicate copy of this sheet is enclosed.
c. ☒ The Director is hereby authorized to charge any additional fees which may be required, or credit any overpayment, to Deposit Account No. 15-0461. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:
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NAME: James A. Oliff
REGISTRATION NUMBER: 27,075

NAME: Joel S. Armstrong
REGISTRATION NUMBER: 36,430

Date: August 28, 2001

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of

Ali HARLIN, Matti HIRVENSALO

Application No.: U. S. National Stage of PCT/FI00/00221

Filed: August 28, 2001 Docket No.: 110486

For: PROCESS FOR PRODUCING A CROSS-LINKED POLYMER PRODUCT

PRELIMINARY AMENDMENT

Director of the U.S. Patent and Trademark Office
Washington, D. C. 20231

Sir:

Prior to initial examination, and after entry of the Annexes to the IPER please amend the above-identified application as follows:

IN THE CLAIMS:

Please replace claims 3-6, 8 and 10-13 as follows:

3. (Amended) A process as claimed in claim 1, **characterized** by also determining the cross-linking degree of the cross-linked polymer product.

4. (Amended) A process as claimed in claim 1, **characterized** by determining the cross-linking degree using a thermomechanical analyzer.

5. (Amended) A process as claimed in claim 1, **characterized** by using a polymer, which is a polyethylene.

6. (Amended) A process as claimed in claim 1, **characterized** by using a grafting agent, which is a silane compound.

8. (Amended) A process as claimed in claim 1, **characterized** by using an initiator, which is a peroxide.

10. (Amended) A process as claimed in claim 1, **characterized** by

using dibutyltin dilaurate as a cross-linking catalyst.

11. (Amended) A process as claimed in claim 1, **w h e r e** the grafted product is a cable or conductor insulation.

12. (Amended) A process as claimed in claim 1, **w h e r e** the grafted product is a pipe.

REMARKS

Claims 1 - 12 are pending. By this Preliminary Amendment, claims 3-6, 8 and 10-12 are amended to remove multiple dependencies. Prompt and favorable examination on the merits is respectfully requested.

The attached Appendix includes marked-up copies of each rewritten claim (37 C.F.R. 1.121(c)(1)(ii)).

Respectfully submitted,

James A. Oliff
Registration No. 27,075

Joel S. Armstrong
Registration No. 36,430

JAO:JSA/cln

Date: August 28, 2001

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DEPOSIT ACCOUNT USE AUTHORIZATION
Please grant any extension necessary for entry;
Charge any fee due to our Deposit Account No. 15-0461

APPENDIX

Changes to Claims:

The following are marked-up versions of the amended claims:

3. (Amended) A process as claimed in claim 1 ~~or 2~~, **characterized** by also determining the cross-linking degree of the cross-linked polymer product.

4. (Amended) A process as claimed in ~~any one of~~ claims 1 ~~to 3~~, **characterized** by determining the cross-linking degree using a thermomechanical analyzer.

5. (Amended) A process as claimed in ~~any one of~~ claims 1 ~~to 4~~, **characterized** by using a polymer, which is a polyethylene.

6. (Amended) A process as claimed in ~~any one of~~ claims 1 ~~to 4~~, **characterized** by using a grafting agent, which is a silane compound.

8. (Amended) A process as claimed in ~~any one of~~ claims 1 ~~to 7~~, **characterized** by using an initiator, which is a peroxide.

10. (Amended) A process as claimed in ~~any one of~~ claims 1 ~~to 8~~, **characterized** by using dibutyltin dilaurate as a cross-linking catalyst.

11. (Amended) A process as claimed in ~~any one of~~ claims 1 ~~to 10~~, **where** the grafted product is a cable or conductor insulation.

12. (Amended) A process as claimed in ~~any one of~~ claims 1 ~~to 11~~, **where** the grafted product is a pipe.

PROCESS FOR PRODUCING A CROSS-LINKED POLYMER PRODUCT

The invention relates to a process for producing a polymer product cross-linked by silane.

It is known in the art that the properties of polymers, such as polyethylene and other polyolefins, can be modified by cross-linking. Polyethylene, for example, can be cross-linked using a peroxide initiator, a hydrolyzing silane compound and a condensation catalyst as described in US patents 3 646 155 and 4 117 195. Said process can be carried out by injecting a polyethylene, a peroxide, for example dicumyl peroxide, a silane compound, for example vinyl trimethoxy silane or vinyl triethoxy silane and a condensation catalyst, for example dibutyltin dilaurate into an extruder and by extruding, whereby a grafted product is obtained which is then processed in condensation conditions in the presence of water or aqueous steam for providing a cross-linked product.

Several drawbacks are frequently associated with prior art cross-linking processes when products of uniform quality are pursued. Such problems occur in particular when the process is continuous. For example, when cable and conductor insulations are formed using a continuous cross-linking process, where the production line is long, a product may be formed which cannot be used owing to the varying quality. The economical losses may be significant in such a case. It is very important to obtain a product of uniform quality particularly when cable and conductor insulations are produced. This obviously holds true also when producing other products, such as pipes, where a continuous process and a long production line are used.

When cable and conductor insulations are concerned the cross-linking degree is of essential importance, as it directly affects the electrical and thermomechanical properties as well as the long-term endurance properties of the product. What mainly affects the cross-linking degree is the amounts of components fed into the extruder, the specific cross-linking conditions, the pressures and temperatures, and also the size of the extruder. For example, when a conductor or cable is insulated by cross-linking polyethylene as shown in the above US patent publications, the amounts of peroxide initiator, silane compound and condensation catalyst affect the cross-linking degree. The insulation properties can be affected by varying said amounts. Until now the cross-linking degree has been manually determined in a laboratory by analyzing a sample taken from the cross-linked product obtained after the condensa-

tion treatment. If the cross-linking degree has been unsatisfactory, the feed of the starting components has been readjusted in order to obtain the desired product. However, the problem is that undesired changes may frequently occur in the reaction conditions of the continuous process that disadvantageously affect the properties of the final product. Since the manual analysis is slow, a lot of poor quality products may be formed that naturally cause economical losses. For example, when cables and conductors are insulated, and the production line is long, the amount of rejected items can be very high, if the feed of the components is adjusted on the basis of the manually obtained analyzing results of the final product.

It should also be pointed out that free silane has a corrosive effect on aluminium conductors in particular. Peroxide residues, in turn, weaken the long-term stability of plastic. Minimizing the residues of both said substances thus improves the long-term endurance properties of the cable.

It has now been found that the above drawbacks can be avoided, if the grafting degree of a polymer is determined at an earlier stage before cross-linking and based thereupon the amounts of substances to be fed into the process are adjusted. The grafting degree of a grafted polymer obtained as an intermediate product in the cross-linking process has to be sufficiently high in order to obtain a high enough cross-linking degree (over 60%) for the final product. By determining the grafting degree using an on line process without disturbing the production process and by continuously adjusting the amounts of starting components on the basis of the result obtained, the quality of the product to be made can be ensured, and the amount of rejected material can be reduced.

The present invention relates to a process for producing a polymer product cross-linked by silane where a polymer, a silane, an initiator and a cross-linking catalyst and possible additives are fed into an extruder and extruded, whereafter the grafted material obtained is cross-linked using water and the catalyst for obtaining a cross-linked polymer product. The process is characterized in that the grafting degree of the grafted material is determined using an on line method and based upon the result obtained the amounts of the components to be fed into the extruder are continuously adjusted in order to obtain the desired grafting degree.

The grafting degree is appropriately determined by measuring the contents of free and grafted silane in the grafted product. This is preferably

carried out with an IR spectrophotometer using a flow through cuvette. The free silane content is determined by measuring the area of the absorption peak at 810 cm^{-1} in the IR spectrum of the silane, whereas the grafted silane content is similarly measured from absorption peak 1080 cm^{-1}. In the calibration known grafted polyethylene silane mixtures are used, whose silane content as well as the silane content of a corresponding grafted material are defined by means of an NMR spectroscope, for example. Since peroxide consumed in the reaction is used as the initiator in polymer silane grafting, the amount of peroxide has to be appropriate, originally about 0.1 % by weight, in order that the silane, the amount of which is about 0.8 to 2 % by weight, is efficiently grafted so that no direct cross-linking disturbing the grafting takes place without silane. In a well-grafted material the amount of remaining peroxide is very low, hardly measurable.

Also the peroxide content can be controlled by measuring the area of the absorption peak at 1155 cm^{-1} by means of an IR spectroscope. The calibration is carried out in the same way as in the case of silane. As the grafting degree is determined using a continuous on line measuring method, the results are rapidly obtained, and the feed of starting components can also be rapidly affected if needed. On the basis of the measuring results the feed of starting components can automatically also be adjusted using a computer as described below.

The adjustment can, for example, be based on an algorithm that may be of a fuzzy logic type. Next the examples of the measurements and adjustments are shown when a silane/peroxide mixture is fed and when silane and peroxide are separately fed.

Example 1 Feeding a ready-mixed silane/peroxide mixture

Measuring (amount)			Control (amount)
grafted silane	free silane	free peroxide	feeding of silane/peroxide mixture
low	low	low	add
low	low	high	add slightly
low	high	low	add slightly
low	high	high	stop

high	low	low	good
high	low	high	reduce slightly
high	high	low	reduce slightly
high	high	high	reduce

Example 2 Feeding silane and peroxide separately

Measuring (amount)			Control (amount)	
grafted silane	free silane	free peroxide	feeding silane	feeding peroxide
low	low	low	add	add
low	low	high	add	-
low	high	low	good	add
low	high	high	stop	stop
high	low	low	good	good
high	low	high	good	reduce
high	high	low	reduce	good
high	high	high	reduce	reduce

Typical polymers to be cross-linked, whose grafting degree can be adjusted by the process of the invention, are polyolefins, preferably polyethylene and polypropylene. The cross-linked product is preferably a cable or conductor insulation, a plastic pipe or profile. The cross-linking catalyst is preferably a dibutyltin dilaurate. A hydrolyzing silane compound, preferably vinyl trimethoxy silane, is used as the grafting agent and the initiator is typically a peroxide compound, preferably dicumyl peroxide. Conventional additives include antioxidants and colourants.

After grafting the material is cross-linked by water and the catalyst. Immediately after the polymer product has been sufficiently cooled the cross-linking degree can further be determined during the run, for example by utilizing a thermomechanical analyzer that measures the elastic properties of the material. When the measuring is carried out the thin measuring head is pressed by a constant load against the cross-linked material and the size of the elastic deflection depending on the cross-linking degree of the polymer, the temperature and the load put upon the measuring head is registered. The registered deflection values are compared with a calibration curve measured

with a corresponding material at the same temperature, thus providing the cross-linking degree corresponding to the deflection. Together the information obtained from the cross-linking degree measurement and the measurement values of the grafting degree and the other measured operating parameters provide feedback to the production process concerning the quality of the final product. The feedback to the production process concerning the quality of the final product makes it substantially easier to find the correct operating parameters for the different raw materials and enables an automatic quality control of the production line.

In its simplest form a thermomechanical analyzer may be composed of two wheels placed at the opposite sides of a cable or another polymer product to be run and pressed against the same, one wheel being pressed by means of a known force, and the deflection caused by the wheel in the product is measured, and of a registration apparatus for registering deflections and also the temperature of the material. The apparatus example shows only one way of measuring the elastic properties of the product on the line during the run, but the example is not intended to restrict the scope of the patent.

CLAIMS (amended on April 12, 2001)

1. A process for producing a polymer product cross-linked by silane where a polymer, a grafting agent, an initiator and a cross-linking catalyst and possible additives are fed into an extruder and extruded, whereafter the grafted material obtained is cross-linked using water and the catalyst for obtaining a cross-linked polymer product, in which process the grafting degree of the grafted material is controlled by an on line method, **characterized** by determining in the flow line after grafting the concentrations of the components affecting the grafting degree and based upon the results obtained, continuously adjusting the amounts of the components to be fed into the extruder in order to obtain the desired grafting degree.

2. A process as claimed in claim 1, **characterized** by determining the concentrations by using IR spectrometry.

3. A process as claimed in claim 1 or 2, **characterized** by also determining the cross-linking degree of the cross-linked polymer product.

4. A process as claimed in any one of claims 1 to 3, **characterized** by determining the cross-linking degree using a thermomechanical analyzer.

5. A process as claimed in any one of claims 1 to 4, **characterized** by using a polymer, which is a polyethylene.

6. A process as claimed in any one of claims 1 to 4, **characterized** by using a grafting agent, which is a silane compound.

7. A process as claimed in claim 6, **characterized** by using a silane compound, which is a vinyl trimethoxy silane.

8. A process as claimed in any one of claims 1 to 7, **characterized** by using an initiator, which is a peroxide.

9. A process as claimed in claim 8, **characterized** by using dicumyl peroxide as an initiator.

10. A process as claimed in any one of claims 1 to 8, **characterized** by using dibutyltin dilaurate as a cross-linking catalyst.

11. A process as claimed in any one of claims 1 to 10, **where** the grafted product is a cable or conductor insulation.

12. A process as claimed in any one of claims 1 to 11, **where** the grafted product is a pipe.

Inventor Information

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Family Name::	HARLIN
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Citizenship Country::	Finland
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Family Name::	HIRVENSALO
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Country of Residence::	Finland
Citizenship Country::	Finland

Correspondence Information

Correspondence Customer Number:: 25944

Application Information

Title Line One::	PROCESS FOR PROCUDING A CROSS-LINKED
Title Line Two::	POLYMER PRODUCT
Total Drawing Sheets::	0
Docket Number::	110486

Continuity Information

>This application is a::	371 of
Application One::	PCT/FI00/00221
Filing Date::	March 17, 2000

Prior Foreign Applications

Foreign Application One::	990613
Filing Date::	March 18, 1999
Country::	Finland
Priority Claimed::	Yes

Assignee Information

Name of assignee::	NEXTROM HOLDING S.A.
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City::	Ecublens/Lausanne
Country::	Switzerland
Postal or Zip Code::	CH-1024

Docket No.: _____

APPLICATION FOR UNITED STATES PATENT
DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name; that

I verily believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural inventors are named below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

PROCESS FOR PRODUCING A CROSS-LINKED POLYMER PRODUCT

described and claimed in the specification:

Check one

*a. ☐ attached hereto.

b. ☒ filed on 17 March 2000 as Application No. PCT/FI00/00221 and amended on 12 April 2001 (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

Under Title 35, U.S. Code §119, the priority benefits of the following foreign application(s) and/or United States provisional application(s) filed by me or my legal representatives or assigns within one year prior to this application are hereby claimed:

990613, Finland, 18 March 1999

The following application(s) for patent or inventor's certificate on this invention were filed in countries foreign to the United States of America either (a) more than one year prior to this application, or (b) before the filing date of the above-named foreign priority application(s) and/or United States provisional application(s):

I hereby appoint the following as my attorneys of record with full power of substitution and revocation to prosecute this application and to transact all business in the Patent Office:

James A. Oliff, Reg. No. 27,075; William P. Berridge, Reg. No. 30,024;
Kirk M. Hudson, Reg. No. 27,562; Thomas J. Pardini, Reg. No. 30,411;
Edward P. Walker, Reg. No. 31,450; Robert A. Miller, Reg. No. 32,771;
Mario A. Costantino, Reg. No. 33,565; Stephen J. Roe, Reg. No. 34,463;
Joel S. Armstrong, Reg. No. 36,430; Christopher W. Brown, Reg. No. 38,025; and
Richard E. Rice, Reg. No. 31,560.

ALL CORRESPONDENCE IN CONNECTION WITH THIS APPLICATION SHOULD BE SENT TO OLIFF & BERRIDGE, PLC, P.O. BOX 19928, ALEXANDRIA, VIRGINIA 22320, TELEPHONE (703) 836-6400.

I hereby declare that I have reviewed and understand the contents of this Declaration, and that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

1 *Typewritten Full Name of First or Sole Inventor*: Ali (Given Name) | [illegible] (Middle Initial) | HARLIN (Family Name)

2 **Inventor's Signature: _____

3 **Date of Signature: August (Month) | 31 (Day) | 2001 (Year)

Residence: Vantaa (City) | _____ (State or Province) | Finland (Country)

Citizenship: Finnish

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*If Box (a.) is checked, this form may be executed only when attached to the specification (including claims).

**Note to Inventor: Please sign name exactly as it appears above and insert actual date of signing.

IF THERE IS MORE THAN ONE INVENTOR USE PAGE 2 AND PLACE AN "X" HERE ☒

PAGE 2 OF U.S.A. DECLARATION FORM
(Discard this page in a sole inventor application)

200

1 *Typewritten Full Name of Second Joint Inventor (if any)*

Given Name	Middle Initial	Family Name
Matti		HIRVENSALO

2 **Inventor's Signature: Matti Hirvensalo

3 **Date of Signature:

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1 *Typewritten Full Name of Third Joint Inventor (if any)*

Given Name	Middle Initial	Family Name

2 **Inventor's Signature:

3 **Date of Signature:

Month	Day	Year

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Citizenship:

Post Office Address: (Insert complete mailing address, including country)

1 *Typewritten Full Name of Fourth Joint Inventor (if any)*

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2 **Inventor's Signature:

3 **Date of Signature:

Month	Day	Year

Residence:

City	State or Province	Country

Citizenship:

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1 *Typewritten Full Name of Fifth Joint Inventor (if any)*

Given Name	Middle Initial	Family Name

2 **Inventor's Signature:

3 **Date of Signature:

Month	Day	Year

Residence:

City	State or Province	Country

Citizenship:

Post Office Address: (Insert complete mailing address, including country)

Note to Inventors: Please sign name exactly as it appears and insert the actual date of signing.

This form may be executed only when attached to the first page of the Declaration and Power of Attorney form of the application to which it pertains.